Use of k_0-INAA at the IJS in CRM production

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16 May 2018
Workshop, ENVCRM,
TUBITAK UME, Gebze, Turkey

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Introduction

Two features of (n,γ) reactor NAA are making its standardization potentially easy and accurate:

1. **the high penetrability of matter for neutrons**
2. **existence of a delayed signal (besides the prompt gamma's).**

Hence, standard and sample can be excited simultaneously and induced signals of both can be measured successively after a suited time following the end of irradiation.

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Introduction

Other consequences:

- NAA is a bulk analysis method with multi-element capability (element concentration and measured signal is nearly matrix-independent).
- Matrix preparation can be kept simple.
- Treatment of sample (and standard) after irradiation is possible (enabling etching, dissolution, chemical separation - RNAA).
- High sensitivity (down to the 10^{-6}, 10^{-9} or even to the 10^{-12} g/g) attainable for many elements.
- Reference method for certification of new CRMs or RMs.

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Introduction

Spectrochimica Acta Part B 66 (2011) 193–241

Contents lists available at ScienceDirect

Spectrochimica Acta Part B

journal homepage: www.elsevier.com/locate/sab

Review

Neutron activation analysis: A primary method of measurement ☆

Robert R. Greenberg [a,*], Peter Bode [b], Elisabete A. De Nadai Fernandes [c]

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ARTICLE INFO

Article history:
Received 12 September 2010
Accepted 23 December 2010
Available online 27 January 2011

Keywords:
Neutron activation analysis
Metrology
Primary method of measurement
Uncertainty budget
Metrological traceability

ABSTRACT

Neutron activation analysis (NAA), based on the comparator method, has the potential to fulfill the requirements of a primary ratio method as defined in 1998 by the Comité Consultatif pour la Quantité de Matière — Métrologie en Chimie (CCQM, Consultative Committee on Amount of Substance — Metrology in Chemistry). This thesis is evidenced in this paper in three chapters by: demonstration that the method is fully physically and chemically understood; that a measurement equation can be written down in which the values of all parameters have dimensions in SI units and thus having the potential for metrological traceability to these units; that all contributions to uncertainty of measurement can be quantitatively evaluated, underpinning the metrological traceability; and that the performance of NAA in CCQM key-comparisons of trace elements in complex matrices between 2000 and 2007 is similar to the performance of Isotope Dilution Mass Spectrometry (IDMS), which had been formerly designated by the CCQM as a primary ratio method.

Published by Elsevier B.V.

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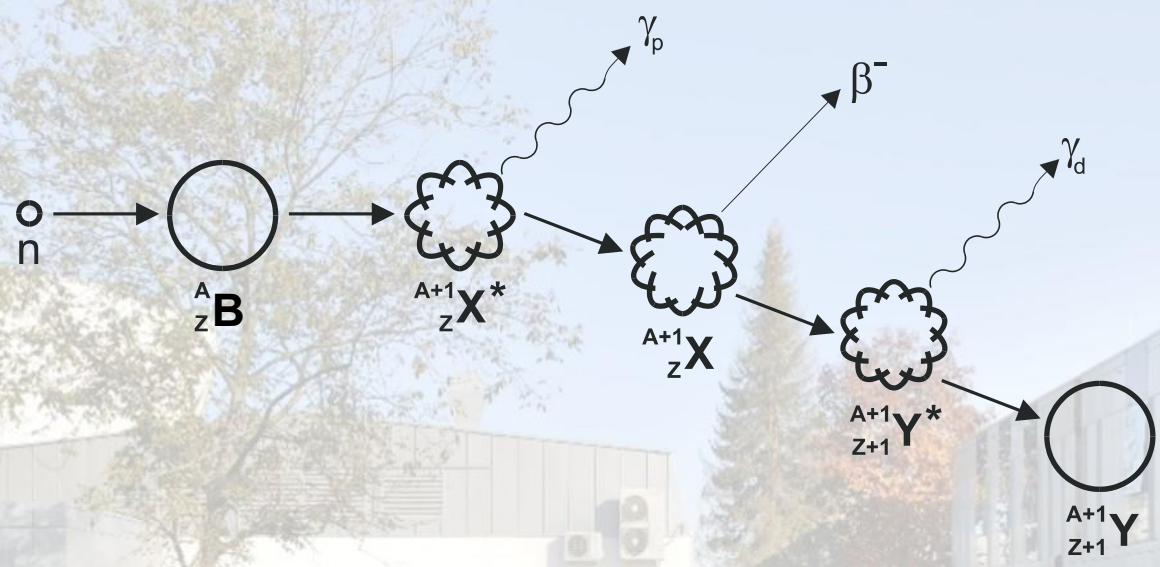
Introduction

Nuclear reactions: - direct (B + a → Y + b); ~ 10^{-22} - 10^{-21} s

- meta stable (B + a → X* → Y + b); ~ 10^{-16} - 10^{-14} s



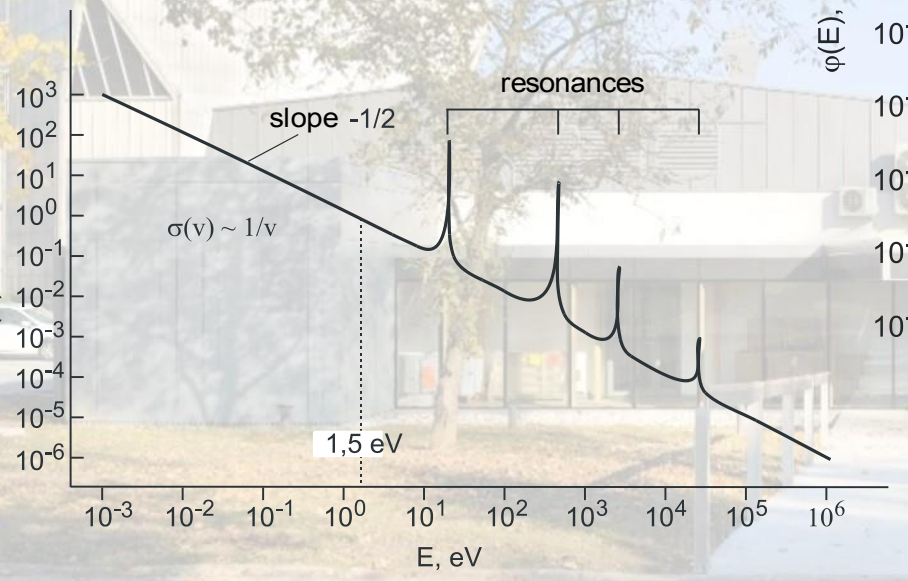
[e. g. $n + {}^{197}_{79}Au \rightarrow {}^{198}_{79}Au^* \rightarrow {}^{198}_{79}Au \rightarrow {}^{198}_{80}Hg^* \rightarrow {}^{198}_{80}Hg$]

A typical (n,γ) reaction with β^- decay.

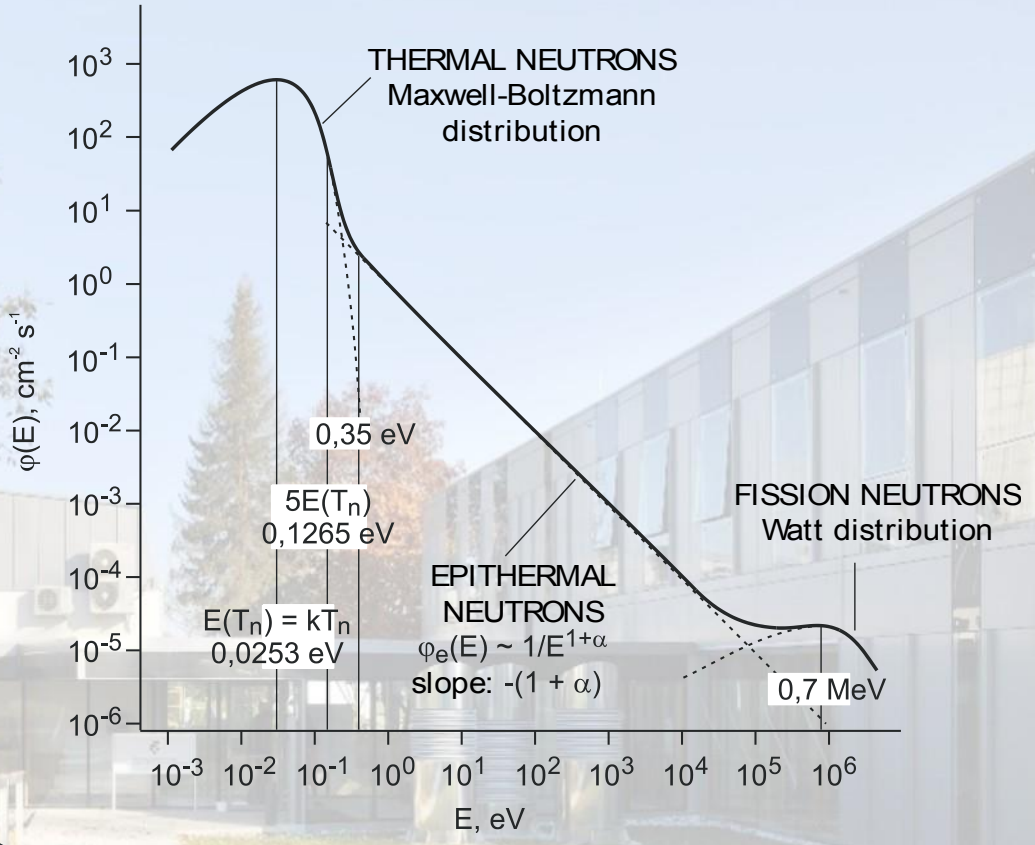
(n,γ) reaction rate

Specific reaction rate per target nuclide

$$R_X = \frac{R}{N_1} = \int_0^\infty \sigma(E)\,\varphi(E)\,dE$$



Cross-section vs. E (σ(v) ~ 1/v)



Neutron flux distribution vs. E

(n, γ) Activation Analysis: principles of standardization

The mass of the element:

$$w_a = \frac{M_a}{N_A \Theta_a \gamma_a} \frac{\left(\frac{N_p / t_m}{S D C}\right)_a}{(G_{th,a} \varphi_{th,a} \sigma_{0,a} + G_{e,a} \varphi_{e,a} I_{0,a}(\alpha)) \varepsilon_{p,a}}$$

- **Relative standardization**
- **Single-comparator standardization**:
  - use of k-factors (experimentally determined)
- **Absolute (parametric) standardization:**
  - condition that φ_{th}, **f** and **α** remain constant during irradiation
  - parameters M, Θ, γ, σ_0 for both taken from literature (accurate known!)
- ***k*$_0$-standardization: KAYZERO/SOLCOI**

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k_0-standardization: KAYZERO/SOLCOI

- k_0-standardization method of NAA was launched in the 1970s
- **SINGCOMP** program: 1987 written for VAX
- **KAYZERO/SOLCOI** program: 1994, 1996, 2003 written for **DOS** and in 2004 written for **Windows**
- Kayzero for Windows (**KayWin®**) ver. 2.42 from March 2011
- KAYZERO library - 144 nuclides (68 elements)
- k_0-NAA became widespread as a practical analytical tool used to analyse different sample matrices
- Kayzero for Windows (**KayWin®**) ver. 3.36 from March 2018

k_0-standardization: KAYZERO/SOLCOI

Thermal and epithermal activation:

$$\rho_a = \frac{\left(\frac{N_p / t_m}{S\,D\,C\,w}\right)_a}{\left(\frac{N_p / t_m}{S\,D\,C\,w}\right)_{Au}} \frac{1}{k_{0,Au}(a)} \frac{G_{th,Au}\, f + G_{e,Au}\, Q_{0,Au}(\alpha)}{G_{th,a}\, f + G_{e,a}\, Q_{0,a}(\alpha)} \frac{\varepsilon_{p,Au}}{\varepsilon_{p,a}}$$

Only epithermal activation:

$$\rho_a = \frac{\left[\left(\frac{N_p / t_m}{S\,D\,C\,w}\right)_{Cd}\right]_a}{\left[\left(\frac{N_p / t_m}{S\,D\,C\,w}\right)_{Cd}\right]_{Au}} \frac{1}{k_{0,Au}(a)} \frac{F_{Cd,Au}\, G_{e,Au}\, Q_{0,Au}(\alpha)}{F_{Cd,a}\, G_{e,a}\, Q_{0,a}(\alpha)} \frac{\varepsilon_{p,Au}}{\varepsilon_{p,a}}$$

k_0-library info:

http://www.kayzero.com/k0naa/k0naaorg/Nuclear_Data_SC/Nuclear_Data_SC.html

J Radioanal Nucl Chem (2014) 300:589–592
DOI 10.1007/s10967-014-3085-2

The 2012 recommended k_0 database

R. Jaćimović · F. De Corte · G. Kennedy · P. Vermaercke · Z. Revay

Received: 31 October 2013 / Published online: 16 March 2014

Abstract Many overview papers have been published with recommended nuclear data for use in the k_0 method of NAA and made available in scientific journals or in the form of a downloadable database. In September 2009, the k_0-International Scientific Committee formed the k_0-Nuclear Data Committee (k_0-NDC) whose first task was to collect all these data at a single place to facilitate updating and to correct any evident errors. This task of the k_0-NDC was successfully completed in March 2012 when the 2012 recommended k_0 database was published in the form of an Excel file.

Keywords k_0 method of NAA · k_0 database · Nuclear data · The IUPAC k_0 database

[1], whereby absolute nuclear data were replaced by k_0 factors, which were experimentally determined. Compared to the relative method, the k_0 method greatly reduces the need for the preparation of standards. It uses gold as the standard and composite nuclear constants for analytically interesting nuclides are normalised to gold nuclear data. During the last 30 years the k_0 method has been introduced in many laboratories around the world for multi-element NAA and the method is continuously improving, along with its nuclear data [2–7]. In 2003, these data were made available by the International Union of Pure and Applied Chemistry (IUPAC) in the form of the Access database (http://www.iupac.org/home/projects/project-db/project-details.html?tx_wfqbe_pi1%5Bproject_nr%5D=2001-075-1-500) created by Kolotov and De Corte [8, 9]. In the process of validation of the consistency of the

Periodic table of the elements (elements in the k_0-library)

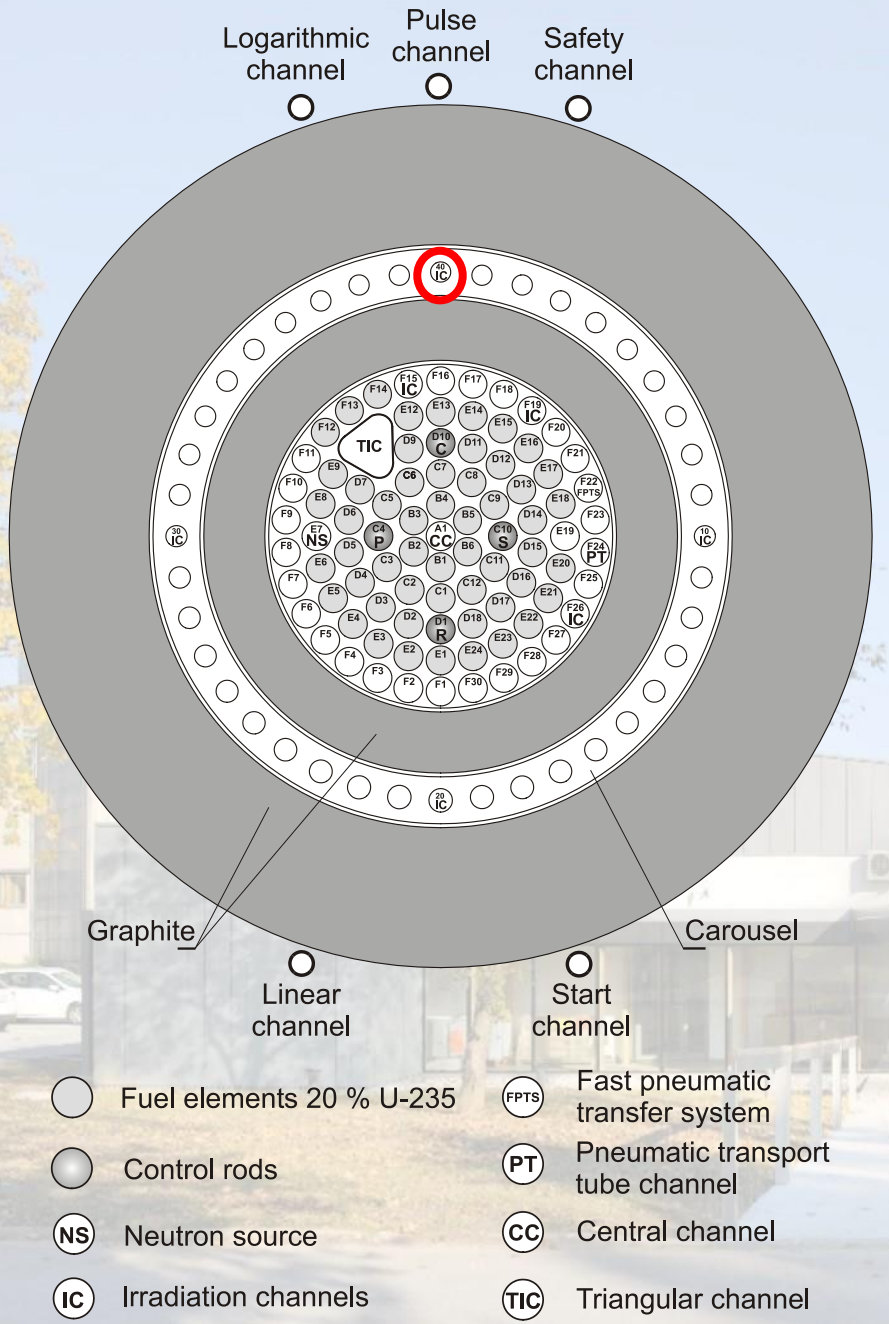
IA	IIA											IIIA	IVA	VA	VIA	VIIA	VIIIA
1 H																	2 He
3 Li	4 Be											5 B	6 C	7 N	8 O	9 F	10 Ne
11 Na	12 Mg	IIIB	IVB	VB	VIB	VIB	{	VIII	}	IB	IIB	13 Al	14 Si	15 P	16 S	17 Cl	18 Ar
19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
55 Cs	56 Ba	57 *La	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn
87 Fr	88 Ra	89 □Ac															

*	58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu
□	90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr

Short irradiation (1-5 min)

Long irradiation (15 - 20 hours)

Westcott factor g ≠ 1



TRIGA Mark II reactor (250 kW)

- Short and long irradiation in the CC:

$$\varphi_{th} \sim 10 \cdot 10^{12}\ cm^{-2}\ s^{-1}$$

- Short irradiation in the PT and in the FPTS (up-to 30 min.)

$$\varphi_{th} \sim 3.5 \cdot 10^{12}\ cm^{-2}\ s^{-1}$$

- Long irradiation in the IC-40 (typically 20 hours)

$$\varphi_{th} \sim 1.1 \cdot 10^{12}\ cm^{-2}\ s^{-1}$$

k_0-INAA analytical procedure

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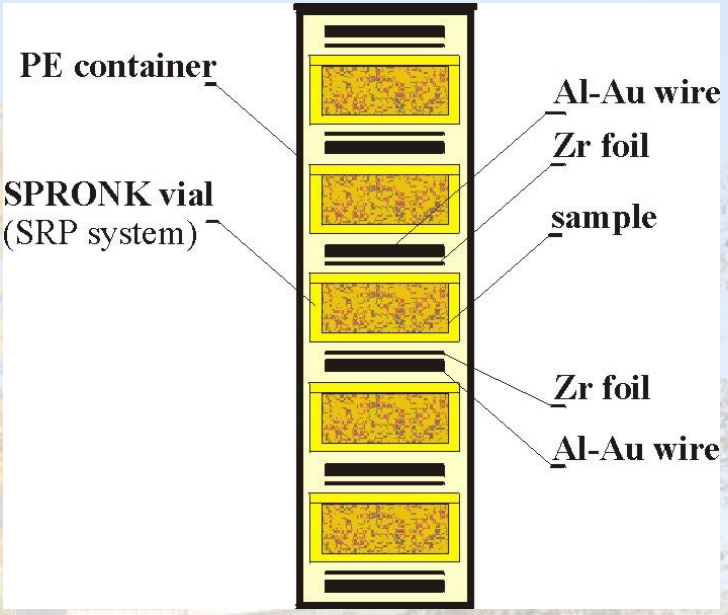
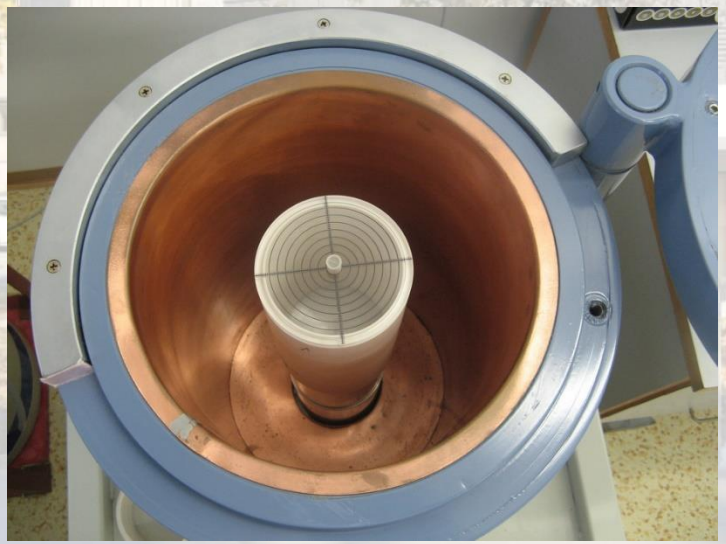
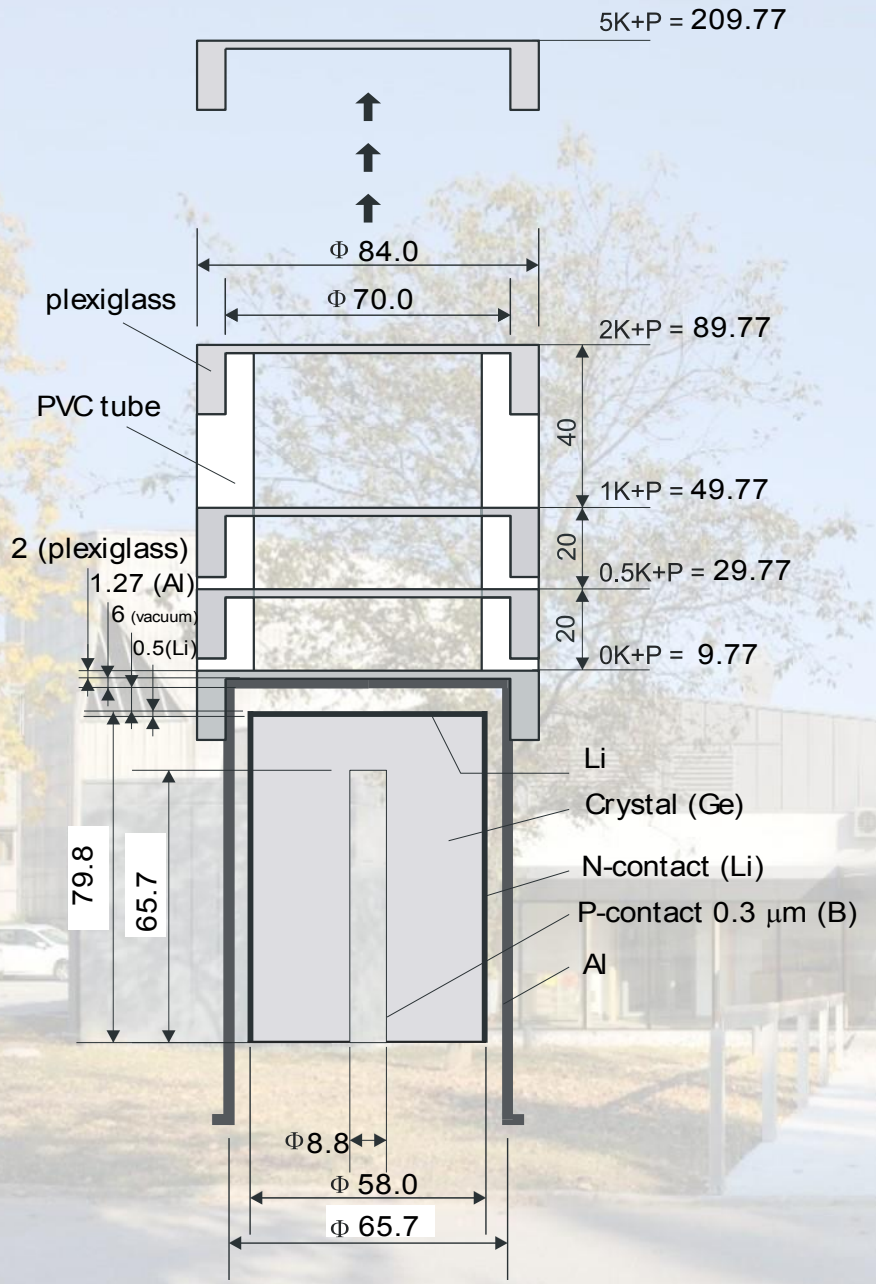




- Sample and standard are prepared in sandwich form and irradiated in the carousel facility of the TRIGA Mark II reactor (250 kW)
- Measurement on an HPGe absolutely calibrated detector
- Evaluation of the spectrum by HyperLab program
- Calculation of the effective solid angle between sample and HPGe detector
- Calculation of element concentration by KayWin®

HPGe detector



HPGe closed end coaxial detector (OR4)
40% relative efficiency at 1332.5 keV (^{60}Co)
("fine tuning" dimensions are in mm)

Advantages and disadvantages to k_0-NAA

- Non-destructive technique which can be applied for different matrices (water, tissues, lichens, aerosols, soils, sediments, sludge's, geological samples, minerals, …)
- k_0-NAA as INAA or RNAA form
- Very small matrix interferences due to behaviour of neutron-sample interactions
- Measuring range from µg/kg up to kg/kg (100%)
- Simultaneously determination of relatively a lot of elements
- Needing absolute calibrated HPGe detector and re-determination of parameters of the neutron spectra when reactor core is change
- Loses of volatilized elements during irradiation (e.g. Hg)
- No speciation for an element
- For some elements limit of detection (LD) is high and RNAA or other techniques should be applied

k_0-NAA quality assessment

Journal of Radioanalytical and Nuclear Chemistry, Vol. 257, No. 3 (2003) 659–663

k_0-NAA quality assessment by analysis of different certified reference materials using the KAYZERO/SOLCOI software

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(Received February 15, 2003)

A suite of natural matrix reference materials (RMs) were used to assess the quality of analytical results obtained by k_0-instrumental neutron activation analysis (k_0-INAA) at the Jožef Stefan Institute (IJS). Five certified reference materials (CRMs) from the Institute for Reference Materials and Measurements (IRMM), two standard reference materials (SRMs) from the National Institute of Standards and Technology (NIST), three RMs from the International Atomic Energy Agency (IAEA) and one RM from IJS were analyzed. Altogether, results for twenty-four elements in inorganic matrices and twenty-nine elements in organic matrices, obtained by k_0-INAA, were compared to certified values. Results obtained show good agreement with certified or assigned values except for Fe, La, Nd, Sm and U in inorganic matrices, and Ag, Al and Cr in organic matrices.

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Participation of IJS/O-2 in certification process of new RMs or CRMs

Organizer	Material	Year	Analytes
INCT	Oriental Basma Tobacco Leaves (INCT-OBTL-5)	2008	As, Au, Ba, Br, Ca, Cd, Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Mo, Na, Nd, Rb, Sb, Sc, Sm, Sr, Ta, Tb, Th, U, Yb, Zn and Zr
INCT	Polish Virginia Tobacco Leaves (INCT-PVTL-6)	2008	As, Ba, Br, Ca, Cd, Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Mo, Na, Nd, Rb, Sb, Sc, Se, Sm, Sr, Ta, Tb, Th, U, Yb, Zn and Zr
INCT	MODAS-2 Bottom Sediment	2013	As, Ba, Co, Cr, Cs, Eu, Fe, Hf, Hg, La, Sb, Sc, Sm, Sr, Tb, Th, U, Zn, Br, Ca, Ce, K, Na, Nd, Rb, Ta, Yb and Zr
IAEA	Determination of Trace Elements in IAEA-452 Biota Sample	2008	Ag, As, Br, Ca, Cd, Co, Cr, Cs, Fe, Hg, K, Mo, Na, Rb, Sb, Sc, Se, Sr and Zn
IAEA	IAEA-CU-2010-02 World-wide open proficiency test: Determination of trace elements in sewage sludge	2010	As, Ba, Co, Cr, Fe, Hg, Se, Sr and Zn
IAEA	Determination of Trace Elements in Marine Biota IAEA-461	2012	Ag, As, Br, Ca, Ce, CH_3Hg, Co, Cr, Cs, Eu, Fe, Hf, Hg, K, La, Mo, Na, Rb, Sc, Se, Sm, Sr, Th, U, Yb and Zn
IAEA	IAEA-MEL CRM Oyster	2013	Ag, As, Br, Ca, CH_3Hg, Co, Cr, Cs, Fe, Hg, K, Na, Rb, Sc, Se, U and Zn
ISPRA	ISPRA RM021 Lagon Sediment	2010	As, Co and Mn
ISPRA	ISPRA RM039 Lake Sediment	2013	As and Co
BAM	BAM YSZO Yttrium Stabilized Zirconium Oxide ERM®-ED105	2013	Th, U and Hf

Participation of IJS/O-2 in certification process of new RMs or CRMs

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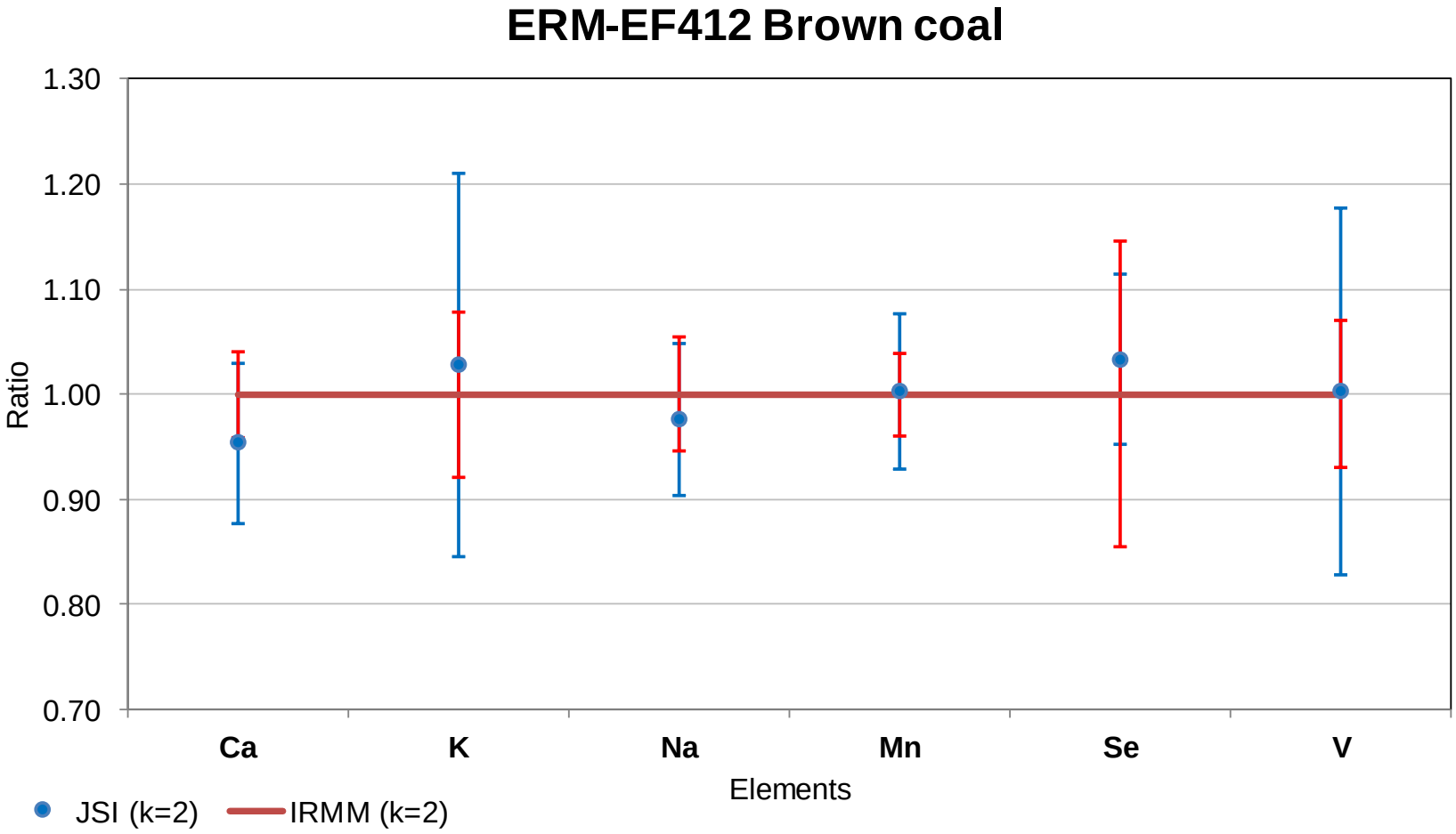
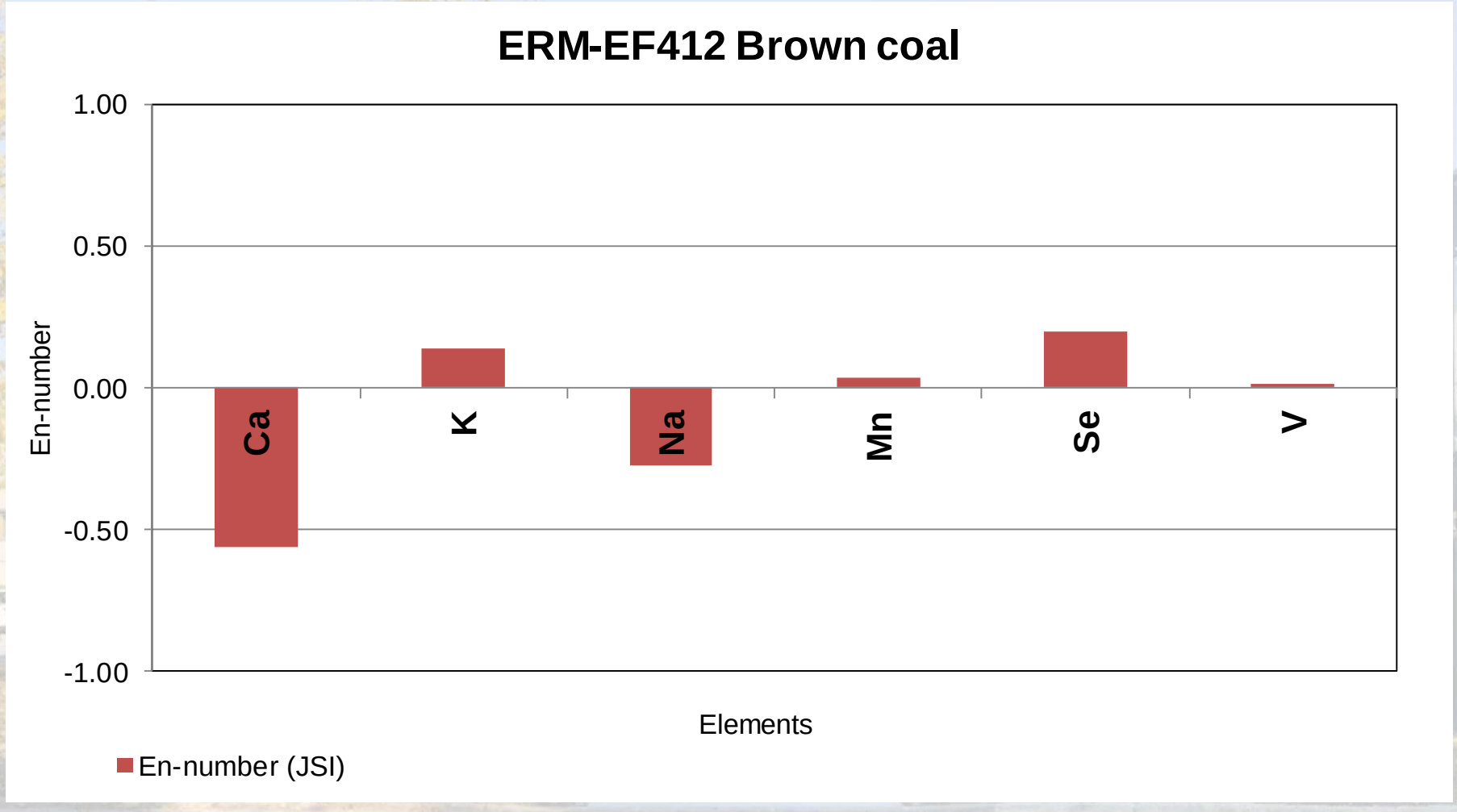
Organizer	Material	Year	Analytes
IRMM	BCR-277R estuarine sediment, BCR-280R lake sediment and BCR-320R channel sediment	2002	Al, As, Ba, Br, Cd, Ce, Cl, Co, Cr, Cs, Cu, Dy, Eu, Fe, Hf, Hg, I, K, La, Mn, Na, Nd, Ni, Rb, Sb, Sc, Se, Sm, Sn, Sr, Ta, Tb, Ti, U, V, Yb and Zn
IRMM	Two polyethylene reference materials ERM®-EC680k and ERM®-EC681k	2006	As, Cd, Cl, Cr, Hg, Sb and Sn
IRMM	Two polymer reference materials ERM-EC590 & ERM-EC591	2008	Br and Sb
IRMM	ERM-CD281 rye grass	2008	As, Cd, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Se, Sn and Zn
IRMM	ERM-CZ120 Fine Dust	2010	As and Cd
IRMM	Total content and aqua regia extractable content of Hg in Loam Soil ERM®-CC141	2010	Hg
IRMM	ERM®-CE278k mussel tissue	2011	Ag, As, Cd, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Rb, Se, Sr and Zn
IRMM	ERM®-DB001human hair	2011	As, Cd, Cu, Hg, Se and Zn
IRMM	ERM®-CD200 Bladderwrack (Fucus vesiculosus)	2011	As, Cd, Hg, Se and Zn
IRMM	ERM®-BD150 and ERM®-BD151 skimmed milk powders	2012	Ca, Cl, K, Mg, Na, Cd, Cu, Fe, Hg, I, Mn, Pb, Se and Zn
IRMM	ERM®-EF411 (hard coal), ERM®-EF412 (brown coal) ERM®-EF413 (furnace coke)	2012	As, Co, Cr, Mn, Sb, Se, V, Zn, Ca, Mg, Na, K, Cl and Hg
IRMM	Determination of trace elements in Lu foil	2013	Major elements and trace elements
IRMM	Mass fraction in Al-0.1%Au alloy: ERM®-EB530A, B and C	2013	Au
IRMM	The minor elements and trace elements mass fraction in TiAl6V4: BCR®-089	2013	Fe, Cr, Mo, Zr, Cu, Co, Mn, W, Zn, Hf, Ta, Hg, La, Ce, Sb, As and Ga

Comparison of k_0-INAA with ERM-EF412 Brown coal



Note: JSI reported also data for As, Co, Cr, Sb, Zn, Mg and Cl obtained by k_0-INAA. For some elements only indicative value were assigned.

Comparison of k_0-INAA with ERM-EF412 Brown coal



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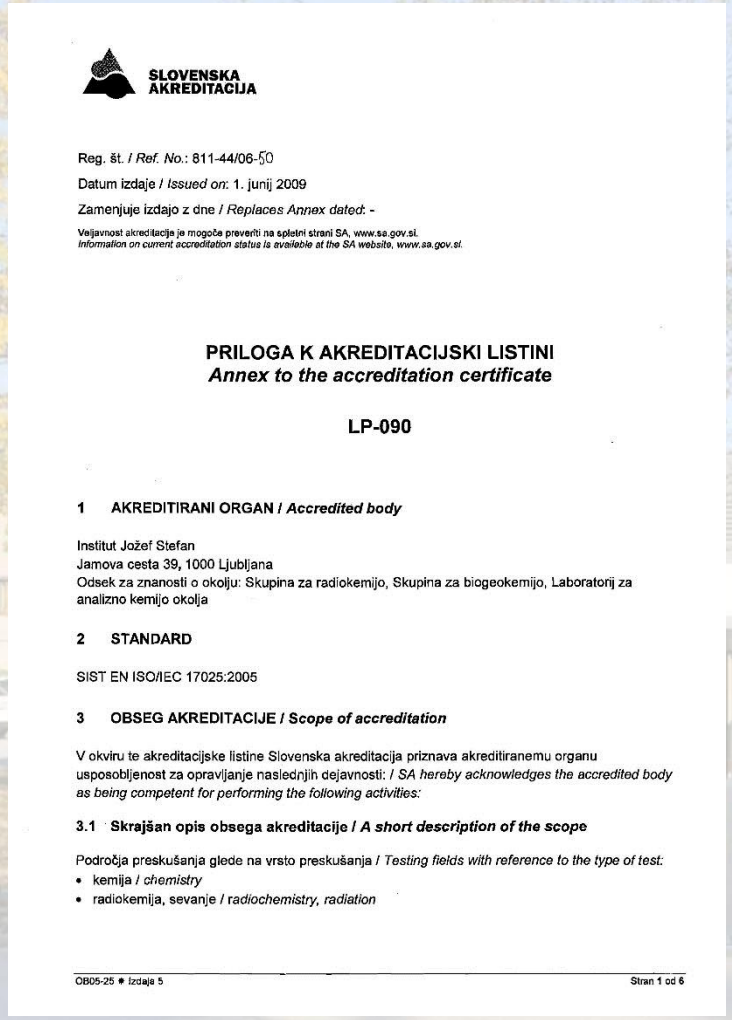
Accreditation Certificate LP-090

2009-06-01



SLOVENSKA AKREDITACIJA

Reg. št. / *Ref. No.*: 811-44/06-50

Datum izdaje / *Issued on*: 1. junij 2009

Zamenjuje izdajo z dne / *Replaces Annex dated*: -

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Annex to the accreditation certificate

LP-090

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Jamova cesta 39, 1000 Ljubljana
Odsek za znanosti o okolju: Skupina za radiokemijo, Skupina za biogeokemijo, Laboratorij za analizno kemijo okolja

2 STANDARD

SIST EN ISO/IEC 17025:2005

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- kemija / *chemistry*
- radiokemija, sevanje / *radiochemistry, radiation*

OB05-25 ● Izdaja 5 Stran 1 od 6

Methods:

1. Determination of **strontium** by beta counting
2. Determination of **tritium activity** by liquid scintillation counting
3. Determination of **^{14}C** in alkaline solution
4. Determining of **elemental composition** of environmental samples using k_0-INAA
5. Determination of **total mercury** in water samples
6. Water quality – Application of inductivity coupled plasma mass spectrometry (ICP-MS) Part 2: Determination of **62 elements**
7. Water quality – Determination of **organotin compounds** – Gas chromatographic method ICP-MS detection

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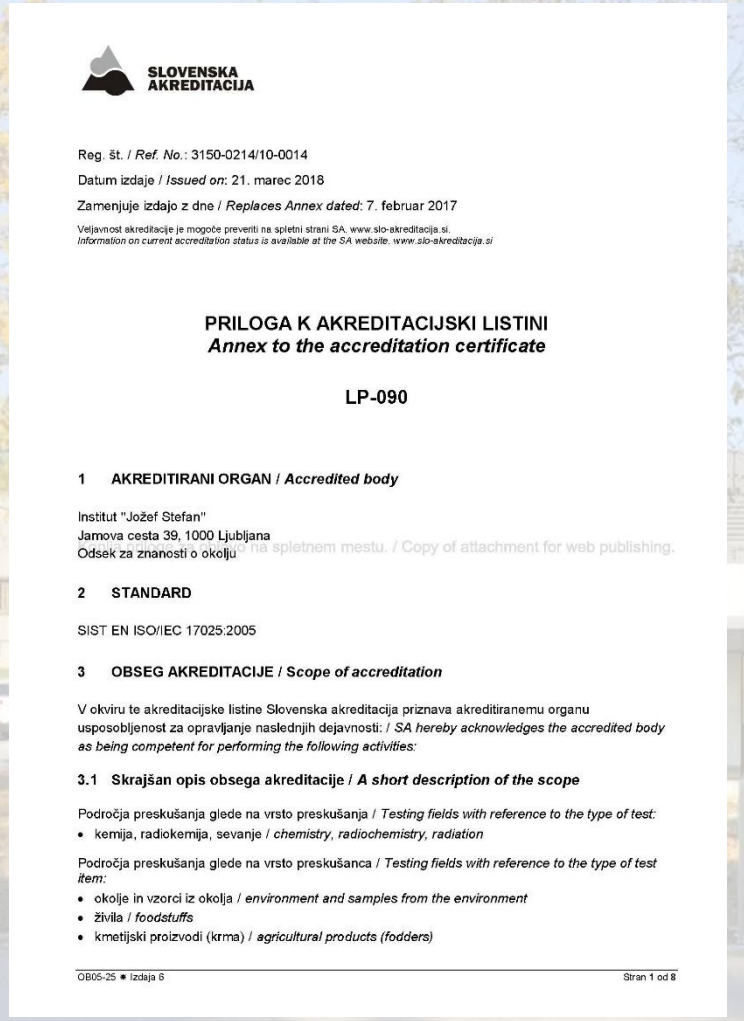
Since 2012



SLOVENSKA AKREDITACIJA

Reg. št. / *Ref. No.*: 3150-0214/10-0014

Datum izdaje / *Issued on*: 21. marec 2018

Zamenjuje izdajo z dne / *Replaces Annex dated*: 7. februar 2017

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- kemija, radiokemija, sevanje / *chemistry, radiochemistry, radiation*

Področja preskušanja glede na vrsto preskušanca / *Testing fields with reference to the type of test item:*
- okolje in vzorci iz okolja / *environment and samples from the environment*
- živila / *foodstuffs*
- kmetijski proizvodi (krma) / *agricultural products (fodders)*

OB05-2S ● Izdaja 6 Stran 1 od 8

Methods:

1. Determination of **strontium** by beta counting
2. Determination of **tritium activity** by liquid scintillation counting
3. Determination of **^{14}C** in alkaline solution
4. Determining of **elemental composition** of environmental samples using k_0-INAA

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Accreditation Certificate LP-090

Scope of the LP-090 dated 2018-03-21:

- **Chemistry**
  - Determination of 31 elements (Ag, As, Au, Br, Ca, Ce, Co, Cr, Cs, Eu, Fe, Hf, Hg, K, La, Mo, Na, Nd, Rb, Sb, Sc, Se, Sm, Sr, Ta, Tb, Th, U, Yb, Zn and Zr) in soil, sediments, ores, sewage sludge, **biological samples, foodstuffs and fuels** by k_0-INAA
- **Radiochemistry**
  - Determination of strontium in samples from the environment (soil, sediment, water, aerosol filter), foodstuffs, milk, feedstuffs and residue by beta counting
  - Determination of tritium activity by liquid scintillation counting (water, urine)
  - Determination of ^{14}C in alkaline solution (water, urine)

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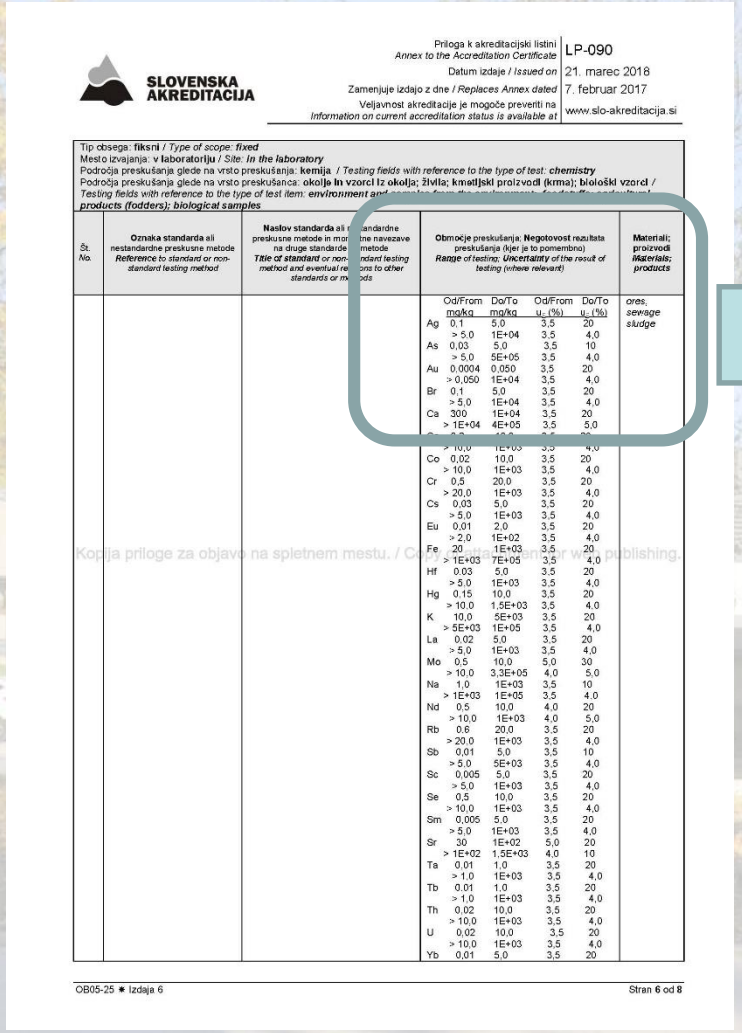
2018-03-21 for k_0-INAA



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Priloga k akreditacijski listini / *Annex to the Accreditation Certificate* LP-090
Datum izdaje / *Issued on* 21. marec 2018
Zamenjuje izdajo z dne / *Replaces Annex dated* 7. februar 2017
Veljavnost akreditacije je mogoče preveriti na / *Information on current accreditation status is available at* www.slo-akreditacija.si

Tip obsega: **fiksni** / *Type of scope: fixed*
Mesto izvajanja: **v laboratoriju** / *Site: In the laboratory*
Področja preskušanja glede na vrsto preskušanja: **kemija** / *Testing fields with reference to the type of test: chemistry*
Področja preskušanja glede na vrsto preskušanca: **okolje in vzorci iz okolja; živila; kmetijski proizvodi (krma); biološki vzorci** / *Testing fields with reference to the type of test item: environment and ... products (fodders); biological samples*

Št. No.	Oznaka standarda ali nestandardne preskusne metode / *Reference to standard or non-standard testing method*	Naslov standarda ali ... preskusne metode in morebitne navezave na druge standarde ali metode / *Title of standard or non-... method and eventual relations to other standards or methods*	Območje preskušanja; Negotovost rezultata preskušanja (kjer je to pomembno) / *Range of testing; Uncertainty of the result of testing (where relevant)*	Materiali; proizvodi / *Materials; products*

	Od/From mg/kg	Do/To mg/kg	Od/From u_c (%)	Do/To u_c (%)
Ag	0,1	5,0	3,5	20
	> 5,0	1E+04	3,5	4,0
As	0,03	5,0	3,5	10
	> 5,0	5E+05	3,5	4,0
Au	0,0004	0,050	3,5	20
	> 0,050	1E+04	3,5	4,0
Br	0,1	5,0	3,5	20
	> 5,0	1E+04	3,5	4,0
Ca	300	1E+04	3,5	20
	> 1E+04	4E+05	3,5	5,0
Ce	0,2	10,0	3,5	20
Co	0,02	10,0	3,5	20
	> 10,0	1E+03	3,5	4,0
Cr	0,5	20,0	3,5	20
	> 20,0	1E+03	3,5	4,0
Cs	0,03	5,0	3,5	20
	> 5,0	1E+03	3,5	4,0
Eu	0,01	2,0	3,5	20
	> 2,0	1E+02	3,5	4,0
Fe	20	1E+03	3,5	20
	> 1E+03	7E+05	3,5	4,0
Hf	0,03	5,0	3,5	20
	> 5,0	1E+03	3,5	4,0
Hg	0,15	10,0	3,5	20
	> 10,0	1,5E+03	3,5	4,0
K	10,0	5E+03	3,5	20
	> 5E+03	1E+05	3,5	4,0
La	0,02	5,0	3,5	20
	> 5,0	1E+03	3,5	4,0
Mo	0,5	10,0	5,0	30
	> 10,0	3,3E+05	4,0	5,0
Na	1,0	1E+03	3,5	10
	> 1E+03	1E+05	3,5	4,0
Nd	0,5	10,0	4,0	20
	> 10,0	1E+03	4,0	5,0
Rb	0,6	20,0	3,5	20
	> 20,0	1E+03	3,5	4,0
Sb	0,01	5,0	3,5	10
	> 5,0	5E+03	3,5	4,0
Sc	0,005	5,0	3,5	20
	> 5,0	1E+03	3,5	4,0
Se	0,5	10,0	3,5	20
	> 10,0	1E+03	3,5	4,0
Sm	0,005	5,0	3,5	20
	> 5,0	1E+03	3,5	4,0
Sr	30	1E+02	5,0	20
	> 1E+02	1,5E+03	4,0	10
Ta	0,01	1,0	3,5	20
	> 1,0	1E+03	3,5	4,0
Tb	0,01	1,0	3,5	20
	> 1,0	1E+03	3,5	4,0
Th	0,02	10,0	3,5	20
	> 10,0	1E+03	3,5	4,0
U	0,02	10,0	3,5	20
	> 10,0	1E+03	3,5	4,0
Yb	0,01	5,0	3,5	20

ores, sewage sludge

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Območje preskušanja; **Negotovost** rezultata preskušanja (kjer je to pomembno)
***Range** of testing; **Uncertainty** of the result of testing (where relevant)*

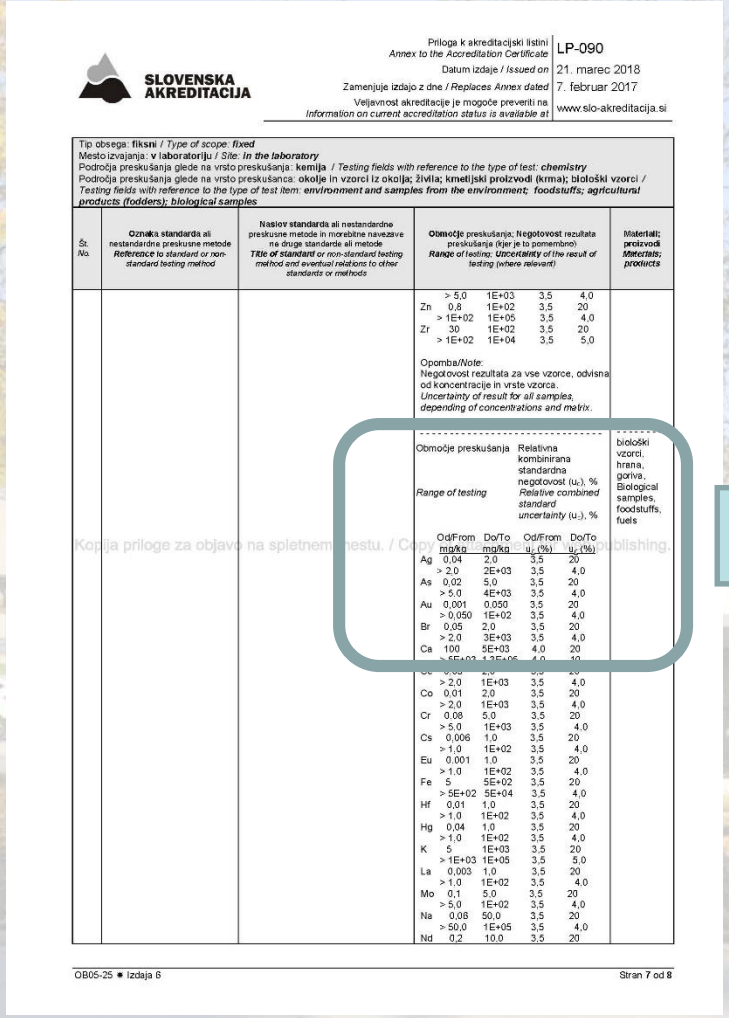
Materiali; proizvodi
Materials; products

	Od/From mg/kg	Do/To mg/kg	Od/From u_c (%)	Do/To u_c (%)
Ag	0,1	5,0	3,5	20
	> 5,0	1E+04	3,5	4,0
As	0,03	5,0	3,5	10
	> 5,0	5E+05	3,5	4,0
Au	0,0004	0,050	3,5	20
	> 0,050	1E+04	3,5	4,0
Br	0,1	5,0	3,5	20
	> 5,0	1E+04	3,5	4,0
Ca	300	1E+04	3,5	20
	> 1E+04	4E+05	3,5	5,0
Ce	0,2	10,0	3,5	20

ores, sewage sludge

Accreditation Certificate LP-090

2018-03-21 for k_0-INAA



SLOVENSKA AKREDITACIJA

Priloga k akreditacijski listini / *Annex to the Accreditation Certificate* LP-090
Datum izdaje / *Issued on* 21. marec 2018
Zamenjuje izdajo z dne / *Replaces Annex dated* 7. februar 2017
Veljavnost akreditacije je mogoče preveriti na / *Information on current accreditation status is available at* www.slo-akreditacija.si

Tip obsega: **fiksni** / *Type of scope: fixed*
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Področja preskušanja glede na vrsto preskušanca: **okolje in vzorci iz okolja; živila; kmetijski proizvodi (krma); biološki vzorci** / *Testing fields with reference to the type of test item: environment and samples from the environment; foodstuffs; agricultural products (fodders); biological samples*

Št. / No.	Oznaka standarda ali nestandardne preskusne metode / *Reference to standard or non-standard testing method*	Naslov standarda ali nestandardne preskusne metode na druga standarda ali metode / *Title of standard or non-standard testing method and eventual relations to other standards or methods*	Območje preskušanja; Negotovost rezultata preskušanja (kjer je to pomembno) / *Range of testing; Uncertainty of the result of testing (where relevant)*	Materiali; proizvodi / *Materials; products*

	> 5,0	1E+03	3,5	4,0
Zn	0,8	1E+02	3,5	20
	> 1E+02	1E+05	3,5	4,0
Zr	30	1E+02	3,5	20
	> 1E+02	1E+04	3,5	5,0

Opomba/*Note:*
Negotovost rezultata za vse vzorce, odvisna od koncentracije in vrste vzorca.
Uncertainty of result for all samples, depending of concentrations and matrix.

Območje preskušanja / *Range of testing*		Relativna kombinirana standardna negotovost (u_c), % / *Relative combined standard uncertainty* (u_c), %		biološki vzorci, hrana, goriva, Biological samples, foodstuffs, fuels
	Od/From mg/kg	Do/To mg/kg	Od/From u_c (%)	Do/To u_c (%)
Ag	0,04	2,0	3,5	20
	> 2,0	2E+03	3,5	4,0
As	0,02	5,0	3,5	20
	> 5,0	4E+03	3,5	4,0
Au	0,001	0,050	3,5	20
	> 0,050	1E+02	3,5	4,0
Br	0,05	2,0	3,5	20
	> 2,0	3E+03	3,5	4,0
Ca	100	5E+03	4,0	20
	> 5E+03	1,3E+05	4,0	10
Co	0,01	2,0	3,5	20
	> 2,0	1E+03	3,5	4,0
Cr	0,08	5,0	3,5	20
	> 5,0	1E+03	3,5	4,0
Cs	0,006	1,0	3,5	20
	> 1,0	1E+02	3,5	4,0
Eu	0,001	1,0	3,5	20
	> 1,0	1E+02	3,5	4,0
Fe	5	5E+02	3,5	20
	> 5E+02	5E+04	3,5	4,0
Hf	0,01	1,0	3,5	20
	> 1,0	1E+02	3,5	4,0
Hg	0,04	1,0	3,5	20
	> 1,0	1E+02	3,5	4,0
K	5	1E+03	3,5	20
	> 1E+03	1E+05	3,5	5,0
La	0,003	1,0	3,5	20
	> 1,0	1E+02	3,5	4,0
Mo	0,1	5,0	3,5	20
	> 5,0	1E+02	3,5	4,0
Na	0,08	50,0	3,5	20
	> 50,0	1E+05	3,5	4,0
Nd	0,2	10,0	3,5	20

OB05-25 ● Izdaja 6 Stran 7 od 8

Območje preskušanja / *Range of testing*		Relativna kombinirana standardna negotovost (u_c), % / *Relative combined standard uncertainty* (u_c), %		biološki vzorci, hrana, goriva, Biological samples, foodstuffs, fuels
	Od/From mg/kg	Do/To mg/kg	Od/From u_c (%)	Do/To u_c (%)
Ag	0,04	2,0	3,5	20
	> 2,0	2E+03	3,5	4,0
As	0,02	5,0	3,5	20
	> 5,0	4E+03	3,5	4,0
Au	0,001	0,050	3,5	20
	> 0,050	1E+02	3,5	4,0
Br	0,05	2,0	3,5	20
	> 2,0	3E+03	3,5	4,0
Ca	100	5E+03	4,0	20
	> 5E+03	1,3E+05	4,0	10

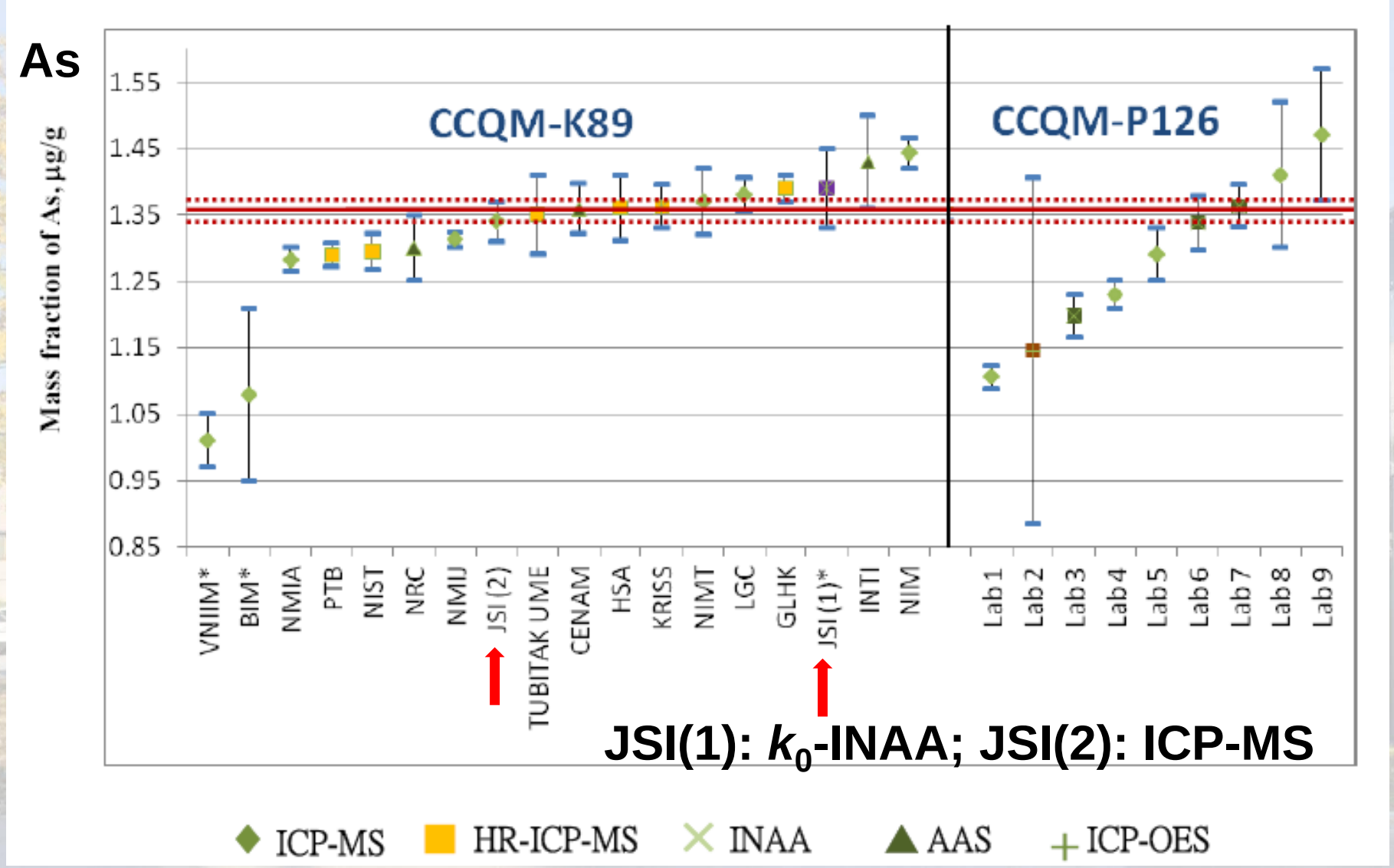
Participation in Inter-comparison studies at the highest level (until 2013)

Institut "Jožef Stefan" Ljubljana, Slovenija

Organizer	Material	Year	Analytes
IRMM	IMEP-14 Sediment	2000	Major and trace elements
	CCQM-K44 Sewage Sludge	2006	Major and trace elements
	IMEP-29 Feed of plant origin	2009	Total As, Cd, Pb, Hg and Sn; Extractable Cd and Pb
	IMEP-112	2011	Total and inorganic arsenic in wheat, vegetable food and algae
	IMEP-38	2013	Determination of total As, Cd, Pb and Hg in compound feed
BAM	CCQM-P34 Aluminum	2002	Fe, Cu, Mn, Cr and Zn
	CCQM-P 34.1 Aluminum	2003	Fe, Cu, Mn, Cr and Zn
	Yttrium Stabilized Zirconium Oxide	2013	Th, U and Hf
IAEA	CCQM-P104 Phosphogypsum	2008	As, Cd, Cr and Cu
	Determination of Trace Elements in IAEA-452 Biota Sample	2008	Trace elements and methylmercury
INCT	INCT-OBTL-5 Oriental Basma Tobacco Leaves	2008	Inorganic trace elements
	INCT-PVTL-6 Polish Virginia Tobacco Leaves	2008	Inorganic trace elements
ISPRA	ISPRA RM021 Lagon Sediment	2010	As, Cd, Ni, Pb, Cu, Co and Mn
	ISPRA RM039 Lake Sediment	2013	As, Cd, Ni, Pb, Cu, and Co
NIM, China	CCQM-P128 & APMP.QM-P17	2011	Pb, As measurements in cosmetic (cream)
	CCQM-K106 & P128.1	2013	Pb, As and Hg measurements in cosmetic (cream)
GLHK	CCQM-K89 & P126	2011	Trace and essential elements in *Herba Ecliptae*
	APMP.QM-S5	2011	Essential and toxic elements in Seafood
CMQ, Chile	CCQM-K30.1 & CCQM-P12.2	2012	Pb in Wine & Pb, Fe, Cu and Cd in Wine
NMIJ	CCQM-K108 & CCQM-P147	2013	Determination of arsenic species, total As and Cd in brown rice flour

CCQM-K89: *Herba Ecliptae*

Institut "Jožef Stefan" Ljubljana, Slovenija



CCQM-K127 and CCQM-P162: Contaminant and other elements in soil: Final Report

M. Rocio Arvizu Torres[1], J. Velina Lara Manzano [1], Milena Horvat [2], Radojko Jaćimović [2], Tea Zuliani [2], Polona Vreča [2]

[1] Centro Nacional de Metrología (CENAM) – Mexico
[2] Jožef Stefan Institute (JSI) - Slovenia

CCQM IAWG Meeting
BIPM, Sèvres, France
24 – 25 April 2017

Bureau International des Poids et Mesures

Home | Key and supplementary comparisons | Calibration and Measurement Capabilities - CMCs

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Key and supplementary comparisons - Information

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CCQM-K127

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Contact us

- BIPM.KCDB@bipm.org

CCQM-K127

Information

Metrology area, branch	Amount of Substance, Inorganics
Description	Toxic and trace elements in soils
Time of measurement	2015
Status	Approved for equivalence, Results available
Reference(s)	*Metrologia*, 2017, **54**, *Tech. Suppl.*, 08010
Measurand	Mass fraction of As, Cd, Fe, Pb and Mn in non-contaminated soil Mass fraction of As, Cd, Fe and Pb in contaminated soil
Transfer device(s)	Dedicated samples
Comparison type	Key comparison
Consultative Committee	CCQM (Consultative Committee for Amount of Substance)
Conducted by	CCQM (Consultative Committee for Amount of Substance)

Top of the page

CCQM-K127: Non-contaminated soil

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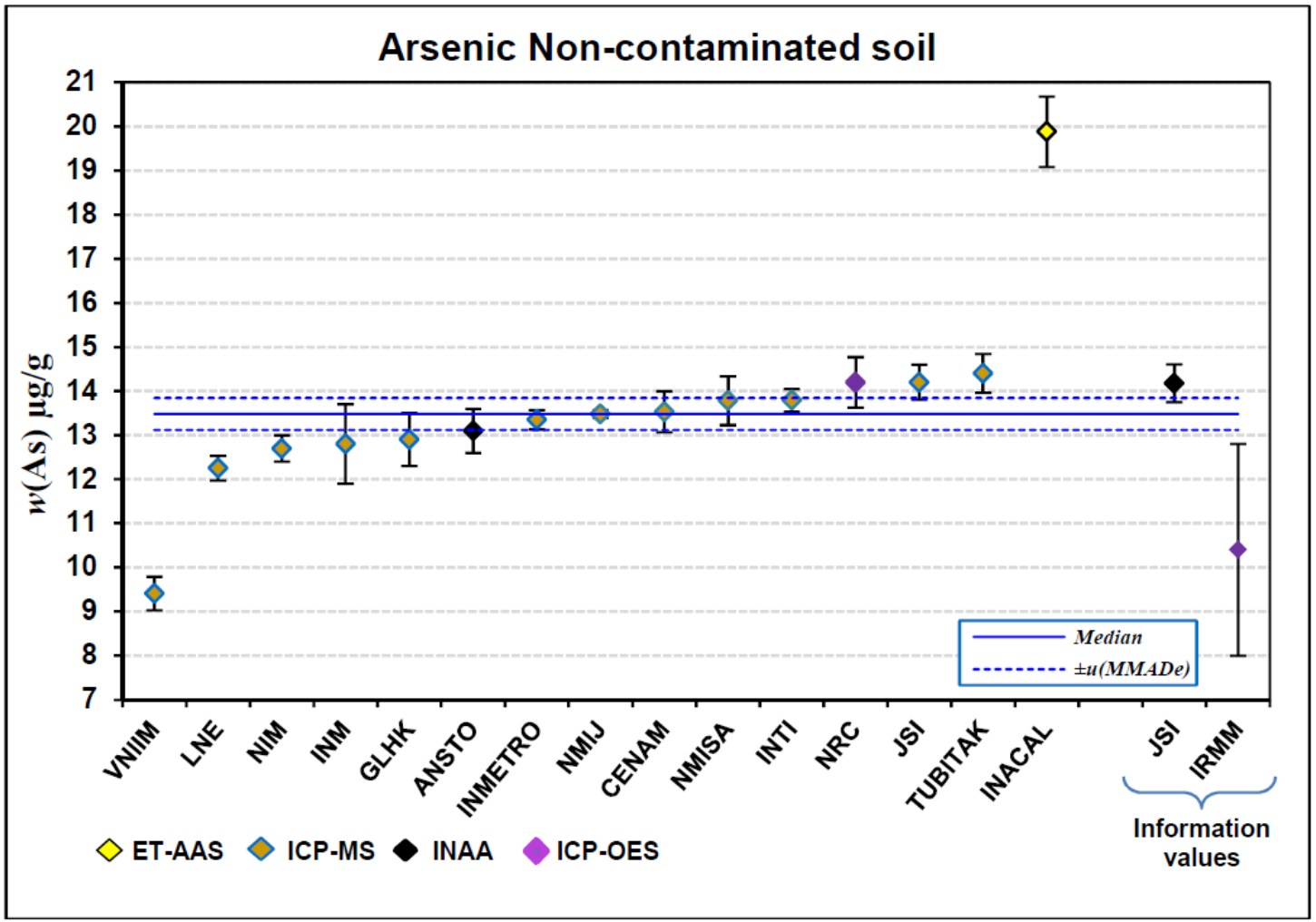


Figure 1. Arsenic in non-contaminated soil and standard uncertainties.
IRMM result was requested to remove.

CCQM-K127: Contaminated soil

Institut "Jožef Stefan" Ljubljana, Slovenija

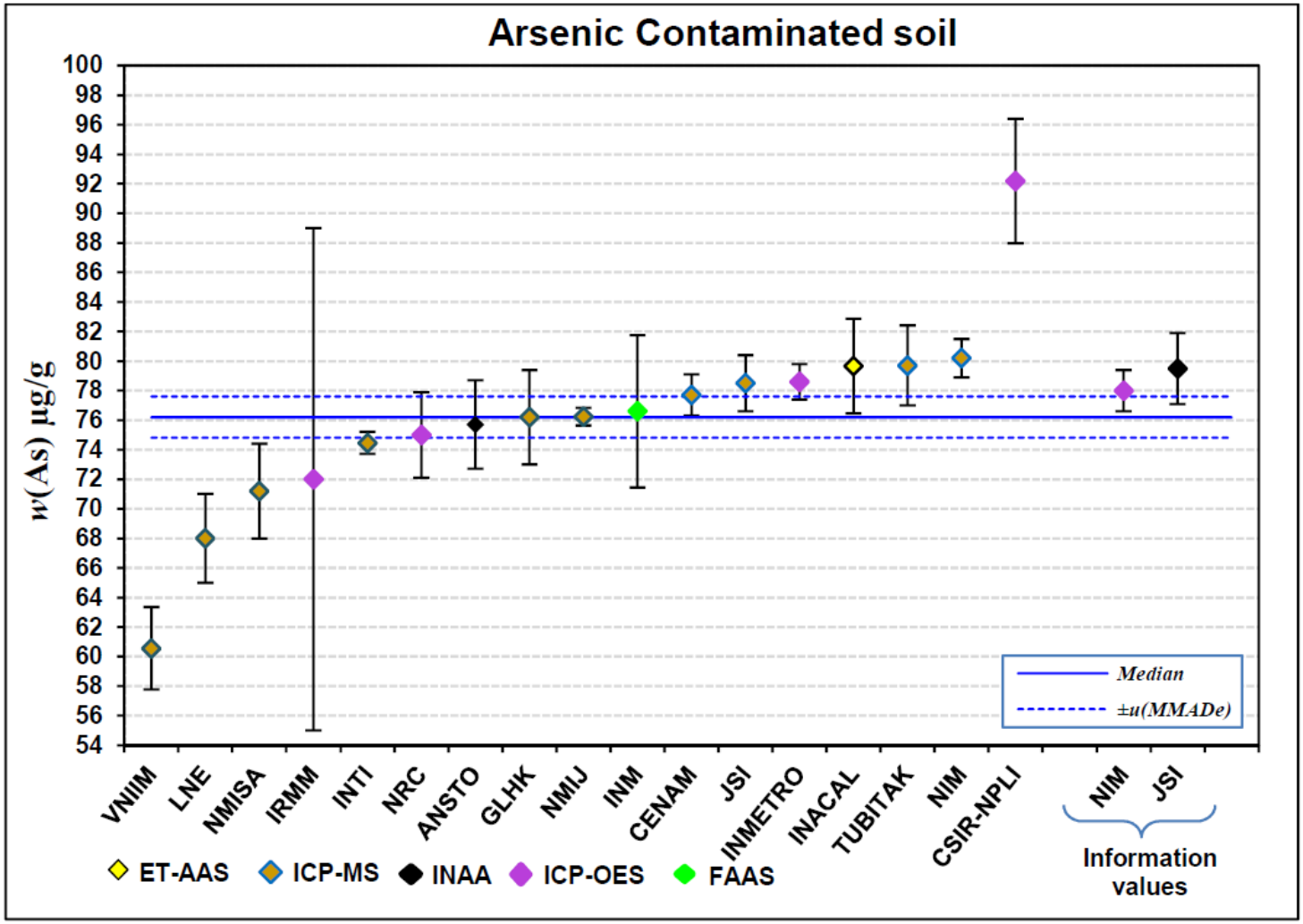


Figure 2. Arsenic in contaminated soil and standard uncertainties.

Calibration and Measurement Capabilities (CMCs) of MIRS/IJS/F-2,O-2

The BIPM key comparison database (2015):

- **Amount of substance:**
  - Category 10 - Biological materials and fluids: **5 CMCs** (As, Ca, Zn, Cd, Pb)
  - Category 11 - Food: **4 CMCs** (As, Zn, Fe, Total As)
  - Category 14 - other materials: **2 CMCs** (As, Hg)

Note: k_0-INAA was used; Category 10 – revised in year 2016

Matrix reference materials for environmental analysis

Short Name: ENVCRM, Project Number: 14RPT03

PARTICIPATING EURAMET NMIS AND DIS

- BAM (Germany)
- DMDM (Serbia)
- GUM (Poland)
- IMBiH (Bosnia and Herzegovina)
- MIKES-SYKE (Finland)
- MIRS/IJS/F-2,O-2 (Slovenia)
- UME (Turkey)

OTHER PARTICIPANTS

National Technical University of Athens - NTUA (Greece)
Uniwersytet Warszawski (Poland)

INFORMATION

TYPE
EMPIR

FIELD
Research Potential

PROJECT TYPE
Joint Research Project

STATUS
in progress

CALL
2014

DURATION
2015 - 2018

Project Partners

EnvCRM

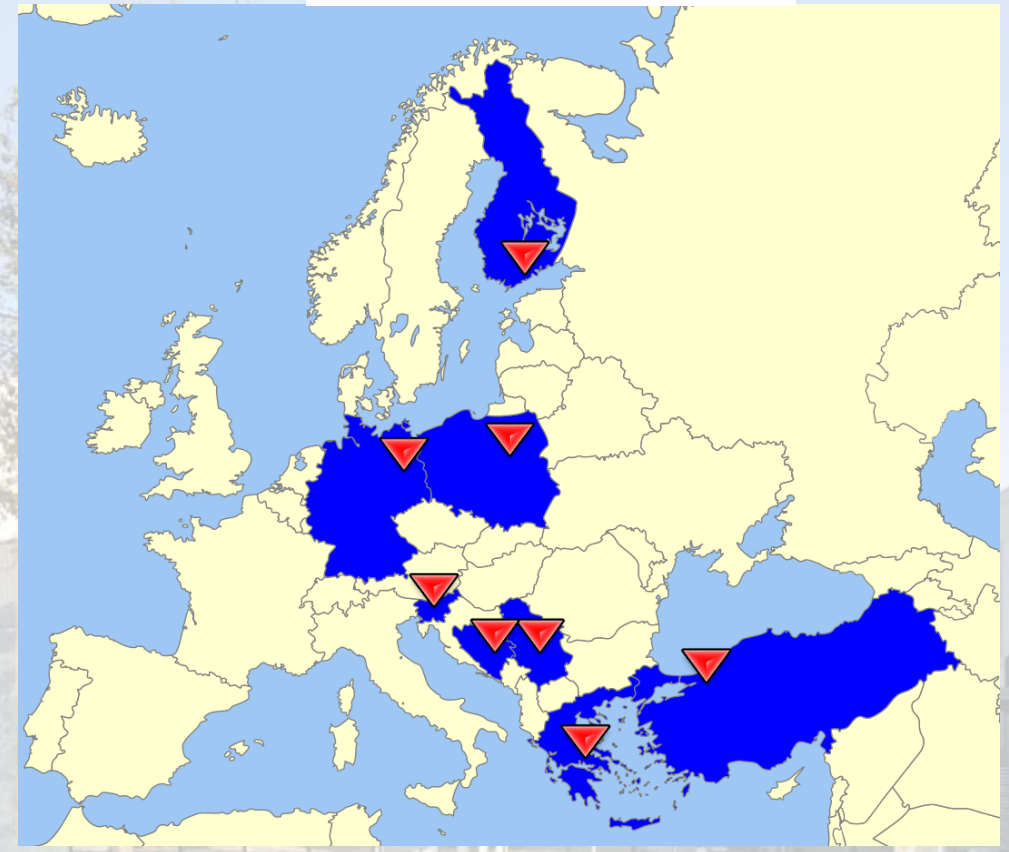
Institut "Jožef Stefan" Ljubljana, Slovenija

EMPIR EURAMET

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

SYKE

Finland



GŁÓWNY URZĄD MIAR GUM

Poland

UNIWERSYTET WARSZAWSKI

BAM

Germany

REPUBLIC OF SERBIA Ministry of economy

DIRECTORATE OF MEASURES AND PRECIOUS METALS

Serbia

IJS

Slovenia

TÜBİTAK UME

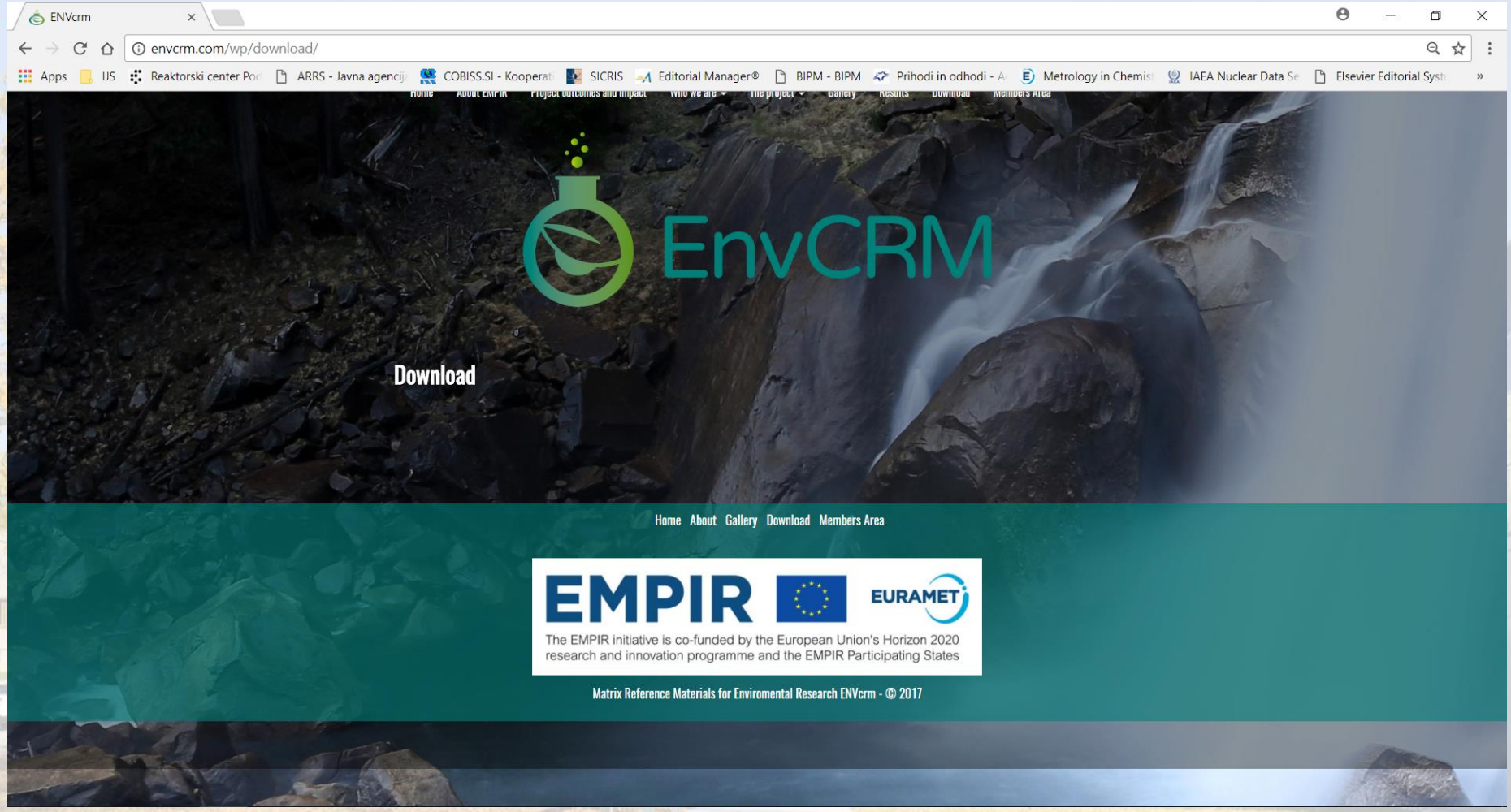
Turkey

IMBIH

Bosnia and Herzegovina

National Technical University of Athens

Greece


ENVcrm
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Apps
IJS
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Matrix Reference Materials for Enviromental Research ENVcrm - © 2017

CRM Candidates

- **Organic ground water CRM candidate**
  - **PFOS (perfluorooctane sulfonate)**
  - **PFOA (perfluorooctanoic acid)**
- **Inorganic water CRM Candidate**
  - **Pb, Cd, Ni, As, Se**
- **Inorganic soil CRM Candidate**
  - **As, Cr, Cd, Hg, Pb, Ni, Fe, Co, Mn, Cu, Zn, V, Sb**

IJS Participation

- **Elements in River Water EnvCRM 02**
  - Homogeneity study
  - Stability study (+18 and +60 ºC):
    - Short-term
    - Long-term
  - **Characterization of Pb, Cd, Ni, As, Se by ICP-MS**
  - **The characterisation was also done under EURAMET.QM-S11 / EURAMET 1424 organized by TUBITAK UME and IMBIH**
- **Elements in soil EnvCRM 03**
  - Homogeneity study
  - Stability study:
    - Short-term
    - Long-term
  - **Characterization of As, Cd, Co, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Sb, V, Zn by ICP-MS, CV AAS and k_0-INAA**

Note: Additionally data for As, Co, Cr, Mn, Sb, V and Zn obtained by ICP-MS were reported.

Acknowledgments

- Prof. F. De Corte, Belgium
- Dr. A. Simonits, Hungary
- Robbert van Sluijs, The Netherlands
- k_0-ISC and k_0-NDSC
- CCQM/IAWG (Dr. Mike Sargent)
- CENAM (M. Rocio Arvizu Torres), Mexico
- ARRS, MIRS
- EMPIR, EVNCRM Project No. 14RPT03 (co-ordinator TUBITAK UME)
- Colleagues at the Department of Environmental Sciences of the Jožef Stefan Institute, Slovenia