

METROLOGY IN CHEMISTRY AT DI MIRS/IJS/O-2

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EURAMET DI workshop, Kongens Lyngby, 18.-19. February, 2016









1949 – Institute of Physics 1959 – Jožef Stefan Nuclear Institute 1969 – Jožef Stefan Institute





●● ● _● Jožef Stefan Institute, Ljubljana, Slovenia



Jožef Stefan 1835 – 1893

The Jožef Stefan Institute is named after the distinguished 19th century physicist Jožef Stefan, most famous for his work on the Stefan-Boltzmann law of black-body radiation

The Mission of the Jožef Stefan Institute: Creation, spread and transfer of knowledge in the fields of natural, life and engineering sciences to the benefit of the society



Human resources:

- 500 researchers
- 250 support staff and administration
- 150 PhD students

CHEMISTRY, BIOCHEMISTRY, NEW MATERIALS

PHYSICS AND NUCLEAR TECHNOLOGY

F1 Theoretical Physics F2 Low and Medium Energy Physics F3 Thin Films and Surfaces F4 Surface Engineering and Optoelectronics F5 Solid State Physics F7 Complex Matter F8 Reactor Physics F9 Experimental Particle Physics R4 Nuclear Engineering K1 Inorganic Chemistry and Technology K3 Physical and Organic Chemistry K5 Electronic Ceramics K6 Engineering Ceramics K7 Nanostructured Materials K8 Synthesis of Materials K9 Advanced Materials B1 –B3 Biochemistry

ENVIRONMENTAL SCIENCE

O2 Environmental Sciences



ELECTRONICS AND INFORMATION TECHNOLOGIES

E1 Automation, Biocybernetics and Robotics E2 Systems and Control E3 Artificial Intelligence E5 Open Computer Systems and Networks E6 Communication Systems E7 Computer Systems E8 Knowledge Technologies E9 Intelligent Systems

Jožef Stefan Institute, Ljubljana, Slovenia

Department of Environmental Sciences <u>www.environment.si</u>

25 researcher; 6 technical and administrative staff, 18 PhD students

- o Environmental analytical chemistry
- Biogeochemical cycling (*including isotope geochemistry*)
- Radiochemistry/radioecology
- Environment and health (*chemicals, food quality, human biomonitoring*)
- Waste treatment, clean technologies
- Modelling, risk assessements & analysis

Am341 Po22 Po23





Funding: 50 % - national research funds 25% - EU funding 25 % - industrial projects

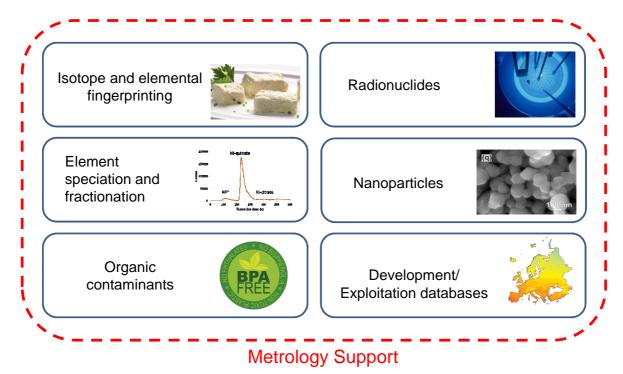








Our research focus...



75 – 85 SCI articles/year; on average 35 citation/article



- Clean laboratories and laboratories for radiochemistry (3000m² laboratory space, 800 m² office space)
- Isotope ratio mass spectrometry EA-IRMS, GC-C-IRMS, DI-IRMS, Py-IRMS, MC-ICP-MS
- Mass spectrometry UPLC-qTOF-MS/MS, ICP-MS, ICP-MS QQQ, LA-ICP-MS, LC-ICP-MS, GC-ICP-MS, SP-ICP-MS, GC(IT)MS, GC-MSD, LC-MS/MS, GC-MS/MS
- Spectrophotometry HG-AFS, CV-AFS
- Nuclear methods

TRIGA MARK II nuclear reactor, alpha, beta and gama counting, NAA

• Access to equipment: JSI departments &infrastructure (microscopy, NMR center)

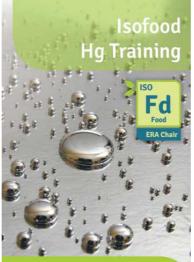
In 2015 opening of the 1100 m² of new and 800 m² of renewed laboratory space with new equipment (6.7 mioEUR)



ERA Chair ISO-FOOD







-

Quality assurance for Hg measurements in food and environmental samples

25th-27th November 2015 Jožef Stefan Institute Ljubljana, Slovenia

www.isofood.eu



ISO-FOOD Summer School Radionuclides in food

June 6-10, 2016 Jožef Stefan Institute, Department of Environmental Sciences, Ljubljana, Slovenia

Objectives



Participant's profile

es. A v

Experience based learning Both theory and practice will be co throughout the course to give participants portunity to put the concepts learned int



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Topics

2. Analytical techniqu natural and man-m

- Separation procedures
- Interpretation of results
- Uncertainty
- Traceability and use of refe



Deadline for application ary 2016 gistration closes 6 May 2016

Candidate selection

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IJS/O-2 – organization of ILCs

- IJS PT-SL1 Sewage sludge (major and trace elements)
- SOIL-1 Preparation and characterization of soil reference material from mercury contaminated site for comparability studies
- PT-WB1, PT-WB2, PT-WB3 Trace elements (Cd, Pb, Hg, As, Se, Cu, Zn) in lyophilized blood
- FF-3613, FF-3614, GG-0461 Trace elements (Cd, Pb, Hg, As, Se, Cu, Zn) in fresh blood
- PT-SED2 Determination of Trace Elements (As, Cd, Co, Cr, Cu, Mo, Ni, Hg, Pb, Zn) in Sediment

: IJS Accreditation Certificate LP-090

2009-06-01

SLOVENSKA AKREDITACIJA

Reg. št. / Ref. No.: 811-44/06-50 Datum izdaie / Issued on: 1. junii 2009

Datum izdaje / issurec or., 1., jung 2000 Zamenjuje izdajo z dne / Replaces Annex dated: -Vejernost ukwatskoje je mogoće preventi na splebni strani 54, www.sa.gov.sl. information or overat accreditatio statu je svabibet a tite 54 webste, www.sa.gov.sl.

PRILOGA K AKREDITACIJSKI LISTINI Annex to the accreditation certific

LP-090

1 AKREDITIRANI ORGAN / Accredited body

Institut Jožef Stefan Jamova ceeta 39, 1000 Ljubljana Odsek za znanosti o okolju: Skupina za radic analizno kemijo okolja kemijo, Skupina za biogeokemijo, Laboratorij za

2 STANDARD

OB06-25 • Izdaja 5

- T EN ISO/IEC 17025:2
- 3 OBSEG AKREDITACIJE / Scope of accreditation

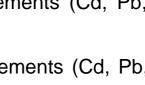
/ okviru te akreditacijske listine Slovenska akreditacija priznava akreditarianemu organu sposobljenost za opravljanje naslednjih dejavnosti: / SA hereby acknowledges the acc to beine company for the sposine the determine of the interview.

3.1 Skrajšan opis obsega akreditacije / A short description of the scope Področja preskušanja glede na vrsto preskušanja / Testing fields with reference to the type of test: kemija / chemistry radiokemija, sevanje / radiochemistry, radiation

Stran 1 od 6

Methods:

- 1. Determination of strontium by beta counting
- 2. Determination of **tritium activity** by liquid scintillation counting
- Determination of ¹⁴C in alkaline solution 3.
- Determining of elemental composition of 4. environmental samples using k_0 -INAA
- Determination of total mercury in water 5. samples
- Water quality Application of inductivity 6. 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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Accreditation Certificate LP-090

Since 2012

Rag. št. / Ref. No.: 811-44/08-50 Datum izdaje / Issued or. 1. junij 2009 Zamenjuje izdajo z dne / Roplaces Annex dated: -Vajernet skretitnicje i mogoče prevent na spolet stara Sk. vene sa gov st. Momination ar cumer sociedanist stabi se zakle at he od te modate, meno sa gov st.

1 AKREDITIRANI ORGAN / Accredited body

3 OBSEG AKREDITACIJE / Scope of accreditati

OB05-25 # Izdaja 5

2 STANDARD SIST EN ISO/IEC 17025:2

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LP-090

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Methods:

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- 4. Determining of **elemental composition** of environmental samples using k_0 -INAA

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: IJS

Accreditation Certificate LP-090 Scope of the LP-090 dated 2014-06-18: • Chemistry

Stran 1 od 6

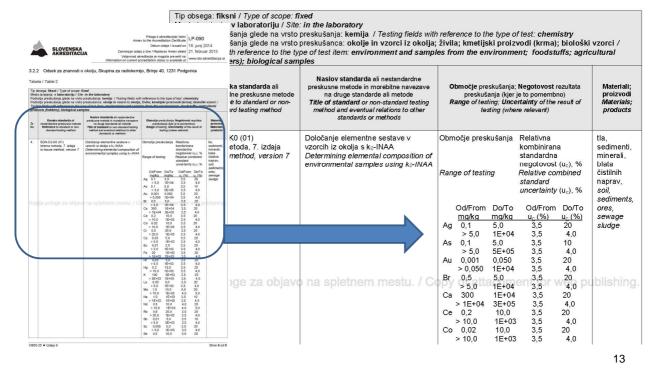
 Determination of 31 elements (Ag, As, Au, Br, Ca, Ce, Co, Cr, Cs, Eu, Fe, Hf, Hg, K, La, Mo, Na, Nd, Rb, Sb, Sc, Se, Sm, Sr, Ta, Tb, Th, U, Yb, Zn and Zr) in soil, sediments, ores, sewage sludge, <u>biological samples, foodstuffs</u> <u>and fuels</u> by k₀-INAA

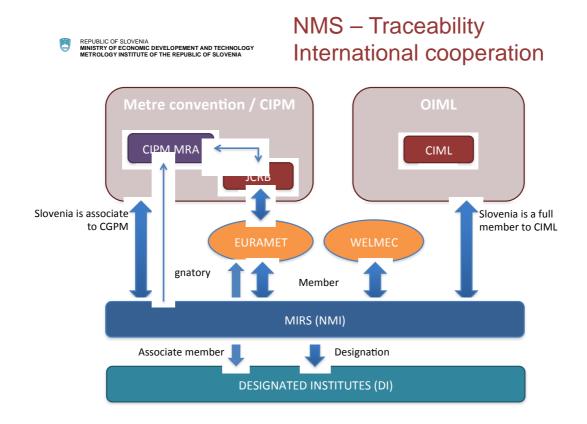
Radiochemistry

- 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 ¹⁴C in alkaline solution (water, urine)

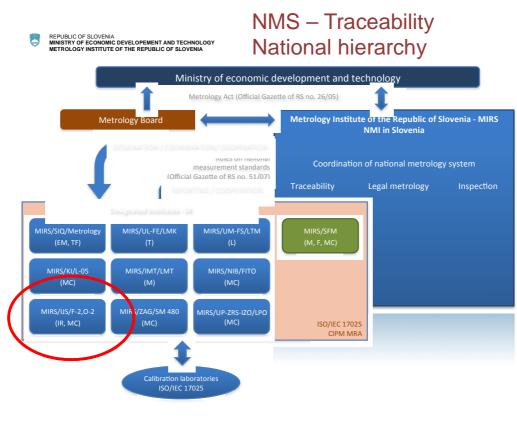
Accreditation Certificate LP-090

2014-06-18 for *k*₀-INAA









MIRS/IJS/O-2

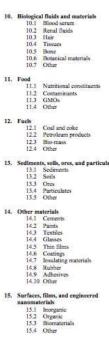
• Designated as DI in 2010, amount of substance (mass fraction of elements): sediments, soils, ores, and particulates !!

No CMCs in KCDB until 2015!!

Why?

			April 2009	
L	High pu	rity chemicals	10,	Biologica
	1.1	Inorganic compounds		10.1
	1.2	Organic compounds		10.2
	1.3	Metals		10.3
	1.4	Isotopics		10.4
	1.5	Other		10.5
				10.6
2.	Inorgan	ic solutions		10.7
	2.1	Elemental		
	2.2	Anionic	11.	Food
	2.3	Other		11.1
				11.2
3.		solutions		11.3
	3.1	PAHs		11.4
	3.2			
	3.3	Pesticides	12.	Fuels
	3.4	Other		12.1
	1000			12.2
4.	Gases			12.3
	4.1	High purity		12.4
	4.2	Environmental	1945	12032
	4.3		13.	
	4.4			13.1
				13.2
	4.6	Other		13.3
- 20				13.4 13.5
5.	Water 5.1			13.5
	5.2	Fresh water Contaminated water	14.	Other ma
	53	Sea water	14.	14.1
	5.4	Other		14.2
	2.4	Other		14.3
6	pH			14.4
	her			14.5
5. 6. 7. 8.	Electrol	vtic conductivity		14.6
03	Lincellion	yne conductivity		14.7
8.	Metal a	nd metal alloys		14.8
	8.1	Ferrous metals		14.9
	8.2	Non-ferrous metals		14.10
	8.3			
	8.4	Other	15.	Surfaces.
		22222	1933	nanomate
9.	Advanc	ed materials		15.1
	9.1	Semiconductors		15.2
	9.2	Superconductors		15.3
	9.3			15.4
	9.4	Ceramics		
	9.5	Other		

LIST OF AMOUNT OF SUBSTANCE CATEGORIES



Participation in Inter-comparison studies at the highest level (until 2013/14)

Organizer	Material	Year	Analytes
	IMEP-14 Sediment	2000	Major and trace elements
	CCQM-K44 Sewage Sludge	2006	Major and trace elements
IRMM	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
	CCQM-P34 Aluminum	2002	Fe, Cu, Mn, Cr and Zn
BAM	CCQM-P 34.1 Aluminum	2003	Fe, Cu, Mn, Cr and Zn
	Yttrium Stabilized Zirconium Oxide	2013	Th, U and Hf
	CCQM-P104 Phosphogypsum	2008	As, Cd, Cr and Cu
IAEA	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	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	ISPRA RM039 Lake Sediment	2013	As, Cd, Ni, Pb, Cu, and Co
NIM,	CCQM-P128 & APMP.QM-P17	2011	Pb, As measurements in cosmetic (cream)
China	CCQM-K106 & P128.1	2013	Pb, As and Hg measurements in cosmetic (cream)
01111/	CCQM-K89 & P126	2011	Trace and essential elements in Herba Ecliptae
GLHK	APMP.QM-S5	2011	Essential and toxic elements in Seafood
CMQ,	CCQM-K30.1 & CCQM-P12.2		Pb in Wine
Chile			& Pb, Fe, Cu and Cd in Wine
NMIJ	CCQM-K08 & CCQM-P147	2013	Determination of arsenic species, total As and Cd in brown rice flour

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MIRS/IJS/O-2

- Since 1968 involved on over 40 certification of matrix reference materials (NIST, IRMM; NIES, IAEA,...)
- Involved in homogeneity testing, characterization and feasibility studies for production of IRMM, IAEA, ISPRA, BAM and INCT reference materials for mass fractions of elements (31 elements) of various origins:
 - Characterization of 18 different RMs during last 4 years e. g. ERM®-EF411 (hard coal), ERM®-EF412 (brown coal) and ERM®-EF413 (furnace coke), ISPRA RM021 (Lagoon Sediment)), etc...
- Participation in national and international projects (also in EMRP ENV02 and EMRP ENV08, ENV052 MeTra)

Co-organization of the CCQM KC



MIRS/IJS/O-2

Problem until September 2014:

 Limited scope of designation in the BIPM database (only amount of substance in sediments, soils, ores, and particulates)

Solution after September 2014:

 Enlarge the scope of designation in the BIPM database to allow the expertise in metrology in chemistry (amount of substance), mass fraction of chemical elements and their species in inorganic and organic matrices



CMC claims in 2014/15 supported by:

- Herba ecliptae CCQM-K89: As, Co, Zn, Cd, Pb
- Seafood APMP.QM-S5: As, Zn, Fe
- Cosmetic cream CCQM-K106: As, Hg
- Brown rice flour: CCQM-K108: As

Conclusions (1/2)

- National metrology system and strategy & goals
 - the role of DIs should be clearly defined (national standards & traceability)
 - prerequisite for sustainable operation of DI and the national metrology systems
 - equality of NMIs and DIs in metrology strategic planning and its implementation
- The area of designation (metrology area)
 - MC should be related to the amount of substance rather than matrix

Conclusions (2/2)

• DI carry important knowledge and expertise and infrastructure

- cost-effective use of resources&expertise

• Effective cooperation/dialog between NMI and DIs and among DIs is needed at the national level

 Allowing for coordinated activities in EURAMET TC's and effective integration of DIs to the activities of EURAMET